

## Topical Symposium on Sustainable Surface Engineering Room Town & Country B - Session TS1-2-MoA

### Coatings for Batteries and Hydrogen Applications II

**Moderators:** Chen-Hao Wang, National Taiwan University of Science and Technology, Taiwan, Martin Welters, KCS Europe GmbH, Germany, Fan-Bean Wu, National United University, Taiwan

2:20pm **TS1-2-MoA-3 Hydrogen-Induced Failure of High-Strength Austenitic Steel Under Wet Friction Conditions, Damian Batory [damian.batory@p.lodz.pl]**, Lodz University of Technology, Poland; Pedro Avila, Etienne Bousser, Thomas Chagnon, Ludvik Martinu, Jolanta Klemberg-Sapieha, Polytechnique Montréal, Canada

This work lies in the combined examination of hydrogen-induced damage and tribological loading, an area where current scientific literature remains largely silent. While the deleterious effects of hydrogen on mechanical properties are well documented, and the fundamentals of friction and wear are extensively studied, the intersection of these two domains has not been systematically explored. By evaluating material responses under simultaneous hydrogen charging and friction-induced stresses, this study provides new insights into the synergistic mechanisms that may govern crack initiation, surface damage evolution, and subsurface microstructural transformations. XM19 austenitic steel samples were subjected to electrochemical hydrogen charging processes before and during tribological testing. After the friction tests a geometry of the obtained wear tracks was analyzed and specific wear rates were calculated. Structure and morphology of the wear tracks were analyzed by SEM and TEM techniques. For the comparison dry and wet friction tests in hydrogen charging solution without electrical potential were also conducted. As the result of the investigation it was noticed that among all charging procedures, the most effective in deterioration of the surface quality after tribological testing was the one with initial precharging and subsequent tribological testing while charging. The post-mortem wear track analysis of these samples revealed the appearance of blisters exhibiting a network of brittle cracks on the surface with elongated island-shaped precipitations of nickel. The size and intensity of blister appearance, as well as the amount of nickel precipitations on the surface of blisters grew with increasing load applied to the friction contact. The cross-section analysis of the blister revealed noticeable changes in the microstructure. Both SEM and TEM analysis of the area directly beneath the blister cavity exhibited signs of severe plastic deformation with markedly fine-grained microstructure and signs of amorphization. The cracks forming the cavity propagate parallel to the surface, however places resembling small voids or cavities connected by the network of cracks oriented one on each other were also registered. A detailed discussion of hydrogen induced failure and nickel precipitation mechanisms is also presented.

2:40pm **TS1-2-MoA-4 Advances in Operando and In Situ Cross-sectional Characterization of Thin Films for Battery and Hydrogen Applications, Juraj Todt [juraj.todt@unileoben.ac.at]**, Montanuniversität Leoben, Austria; Francois Lienard, Manfred Burghammer, ESRF, Grenoble, France; Tobias Huber, Huber Scientific, Austria; Henrik Bratlie, Daniel Rettenwender, Norwegian University of Science and Technology (NTNU), Norway; Rostislav Daniel, Markus Alfreider, Michael Tkadletz, Jozef Keckes, Montanuniversität Leoben, Austria

#### INVITED

As we are facing the worldwide need to reduce the carbon footprint of modern technologies, materials relevant to the fields of sustainable energy storage and transport have received widespread attention. A great deal of research effort has been devoted to their study and optimization, where current techniques often measure either bulk or aggregate properties such as specific capacities or power densities. However, to fully understand all the intricate and often localized mechanisms at play, a more detailed look is necessary. Methods such as impedance spectroscopy are able to capture the crucial role of interfaces with the help of some modeling, but direct observations for cross-sectional behavior are usually lacking.

This is where cross-sectional X-ray nano-diffraction (CSnanoXRD) could provide a very effective tool, as it offers the capability of comprehensive insights encompassing phase composition, preferred crystallite sizes and orientations, accumulated lattice defects and crucially also information on internal strains and stresses. This contribution aims to present our recent methodological advances in applying the CSnanoXRD concept to battery and hydrogen applications, showcasing the dedicated experimental set-ups that are necessary to make this possible.

For the study of zero-excess solid state batteries, a test platform capable to apply stacking pressures in the range of several 100 MPa on X-ray-transparent cross-sectional lamellae has been developed within the Horizon Europe project OPERA. First results on the role of anode interlayers in Li and Na deposition at the interface between solid electrolytes and current collectors will be shown. A focus will lie on the many technological challenges that had to be overcome until a fully working tool has been achieved, which is now available to the scientific community.

A further novel test platform will be presented, aimed at the study of hydrogen interaction with surface layers. It is based on the application of femtosecond laser ablation for sample patterning and two-photon lithography to create the appropriate microfluidic structures necessary for electrolytic H-charging of thin films at a CSnanoXRD experimental station. The behavior of various metallic layers including Ni and V, as well as Pd/Nb and Ti-V will be examined in detail, showing the formation of hydride phases and decoupling lattice expansion due to H uptake from the formation of residual stress due to mechanical constraints from the substrate and underlying material. Future possibilities for this approach will comprise the study of the role of interfaces and multilayered H-barrier structures.

3:20pm **TS1-2-MoA-6 Atomic Layer Deposition for Enhancing Durability of Fuel Cell Catalysts, Shao-Chuan Chang, Chih-Liang Wang [wangcl@mx.nthu.edu.tw]**, Department of Materials Science and Engineering, National Tsing Hua University, Taiwan

Fuel cells are promising clean energy devices for hydrogen conversion, yet their commercialization is hindered by the high cost and limited durability of catalysts. To address these challenges, developing low-cost alternatives with enhanced stability is essential. Surface coating has emerged as an effective strategy to improve catalyst durability by suppressing metal particle agglomeration, dissolution, and carbon corrosion. In this study, Ag/C catalysts were prepared via a microwave-assisted method and subsequently coated with zinc oxide (ZnO) using atomic layer deposition (ALD) to evaluate their stability for anion exchange membrane fuel cell (AEMFC) applications. Ag/C catalysts obtained by conventional impregnation were also investigated for comparison. Structural (XRD, FTIR, TEM) and electrochemical (CV, ECSA) analyses demonstrated that the ALD ZnO-coated Ag/C prepared via the microwave-assisted route possessed markedly enhanced durability relative to the impregnated counterpart. Single-cell performance tests further confirmed the superior activity of the microwave-assisted ZnO-coated Ag/C catalyst, which achieved a higher peak power density than the impregnated sample. These results confirm that an optimally engineered ALD ZnO coating effectively mitigates Ag particle aggregation and dissolution, thereby stabilizing the catalyst structure and enhancing overall AEMFC performance.

4:00pm **TS1-2-MoA-8 Electrocatalytic Performance Analysis of FeNiMoWCu High Entropy Alloy Thin Films: Effects of Ni Content, Yen-Chin Lai [lai.juju0120@gmail.com]**, Po-Chun Chen, National Taipei University of Technology, Taiwan; Bih-Show Lou, Chang Gung University, Taiwan; Jyh-Wei Lee, Ming Chi University of Technology, Taiwan

Hydrogen energy has attracted significant attention due to its cleanliness, non-polluting and carbon-free characteristics [1]. Among various hydrogen production methods, water splitting is considered one of the most promising. However, both the hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) require catalysts to reduce energy losses caused by overpotential. Currently, the most widely used electrocatalysts are based on expensive noble metals, such as platinum and palladium [2], which limits their large-scale application. Therefore, this study aims to develop low-cost, stable, and highly efficient FeNiMoWCu high entropy alloy (HEA) thin films as potential electrocatalyst materials for water-splitting applications [3].

In this research, equimolar FeNiMoWCu targets and pure Ni targets were employed to deposit FeNiMoWCu high entropy alloy (HEA) thin films with different nickel contents onto 304 stainless steel, silicon wafers, and nickel foam (NF) substrates using a co-sputtering system. Grazing-incidence X-ray diffraction (XRD) analysis revealed that all films exhibited a typical amorphous structure. The nickel content has no influence on the crystal structure of thin films. The cross-sectional morphologies indicated that all films possessed dense microstructures without noticeable defects. Mechanical property measurements demonstrated stable hardness values, while scratch testing confirmed excellent adhesion, with all films showing a critical load ( $L_{c3}$ ) exceeding 100 N.

Lower overpotential results than untreated NF for the evolution reaction (HER) were obtained for the FeNiMoWCu thin films deposited on NF after

the electrochemical test in 1 M KOH aqueous solution. The effect of Ni content on the electrocatalysis performance of HEA thin films was explored. We can conclude that the HEA film prepared at a Ni target power of 75 W exhibited the best HER performance, with an overpotential of 121 mV and a Tafel slope of 177.7 mV/dec at a current density of 10 mA/cm<sup>2</sup>, indicating excellent electrocatalytic activity for hydrogen evolution. This research develops the novel FeNiMoWCu electrocatalyst thin film materials for water-splitting hydrogen production. The results will provide insights into the potential of FeNiMoWCu HEA films for green energy applications with enhanced electrocatalytic performance.

Keywords: High entropy alloy thin films, Hydrogen evolution reaction, FeNi<sub>x</sub>MoWCu, electrocatalytic property

4:20pm **TS1-2-MoA-9 Development of FeNiMoWCuN and FeNiMoWCu High Entropy Alloy Thin Film as Efficient Electrocatalysts for Water-splitting Applications**, *TAI Kao Cheng [dylan37859631@gmail.com], Lee Jyh-Wei*, Ming Chi University of Technology, Taiwan; *Lou Bih-Show*, Chang Gung University, Taoyuan, Taiwan; *Li Chia-Lin*, Ming Chi University of Technology, Taiwan

This study investigates the microstructure and electrocatalytic properties of FeNiMoWCuN and FeNiMoWCu high entropy alloy (HEA) thin films deposited by high power impulse magnetron sputtering (HiPIMS) and direct current (DC) magnetron sputtering techniques, respectively. The HEA films were fabricated at various argon-to-nitrogen and argon-to-acetylene gas flow ratios to assess the impact of nitrogen and carbon contents, respectively, on the phase, microstructure, and electrocatalytic properties of the thin films. A proper nitrogen content was found to promote the formation of the metal nitride phase, thereby enhancing the electrocatalytic activity of the films. Notably, improved performance was observed for the HEA films with different N contents in the hydrogen evolution reaction (HER) and oxygen evolution reaction (OER), characterized by lower overpotentials and smaller Tafel slopes. Similarly, a proper carbon content was achieved for FeNiMoWCu, resulting in improved electrocatalytic performance. Overall, FeNiMoWCuN and FeNiMoWCu thin films deposited using HiPIMS and DC with optimized nitrogen content exhibited superior electrocatalytic properties, highlighting their potential for applications in water splitting.

4:40pm **TS1-2-MoA-10 High-Entropy Oxide Thin Films for Sustainable Battery Applications**, *Pavel Soucek [soucek@physics.muni.cz], Tatiana Pitonakova, Tomas Rada*, Masaryk University, Czechia; *Tomas Kazda, Antonin Simek*, Brno University of Technology, Czechia; *Petr Vasina*, Masaryk University, Czechia

#### INVITED

High-entropy materials represent a rapidly growing frontier in materials science, offering new routes toward multifunctional and resource-efficient technologies. While high-entropy alloys (HEAs) have gained considerable attention, the concept has now been successfully extended to ceramics, including oxides, nitrides, borides, and carbides. These materials derive their unique properties from four synergistic effects: high configurational entropy, severe lattice distortion, sluggish diffusion, and the cocktail effect. The resulting structural stability, chemical resilience, and tunable electronic properties make high-entropy oxides (HEOs) highly promising candidates for advanced electrochemical applications such as lithium-ion and sodium-ion batteries.

In this contribution, we present a study of the Mg-Co-Ni-Cu-Zn-O system, crystallizing in a rock salt-type (MgCoNiCuZn)O structure, and Cr-Mn-Fe-Ni-Cu-O system, crystallizing in a spinel-type (CrMnFeNiCu)<sub>4</sub>O<sub>4</sub> structure. While the first system represents a “conservative” choice close to current materials used in batteries, the second is entirely free of critical raw materials while offering the potential for improved electrochemical performance and long-term stability, aligning with the vision of sustainable and circular energy storage technologies. The thin films are deposited by reactive DC and High Power Impulse Magnetron Sputtering (HiPIMS), allowing precise control of elemental composition and plasma energy input. The influence of deposition temperature, stoichiometry, and process parameters on the morphology and crystalline structure of the as-deposited coatings is systematically investigated.

Special emphasis is placed on the fabrication of porous thin films, achieved by adjusting the deposition pressure, target-to-substrate distance, and substrate tilt angle—from normal to glancing-angle configurations. The resulting morphological variations strongly affect ion transport and electrochemical activity. Finally, the electrochemical behavior of selected coatings against lithium and sodium is evaluated and correlated with their structural and compositional features. The findings open new pathways for designing high-entropy oxide electrodes that combine sustainability,

structural tunability, and superior performance for next-generation battery systems.

## Author Index

**Bold page numbers indicate presenter**

**— A —**

Alfreider, Markus: TS1-2-MoA-4, 1  
Avila, Pedro: TS1-2-MoA-3, 1

**— B —**

Batory, Damian: TS1-2-MoA-3, **1**  
Bih-Show, Lou: TS1-2-MoA-9, 2  
Bousser, Etienne: TS1-2-MoA-3, 1  
Bratlie, Henrik: TS1-2-MoA-4, 1  
Burghammer, Manfred: TS1-2-MoA-4, 1

**— C —**

Chagnon, Thomas: TS1-2-MoA-3, 1  
Chang, Shao-Chuan: TS1-2-MoA-6, 1  
Chen, Po-Chun: TS1-2-MoA-6, 1  
Chia-Lin, Li: TS1-2-MoA-9, 2

**— D —**

Daniel, Rostislav: TS1-2-MoA-4, 1

**— H —**

Huber, Tobias: TS1-2-MoA-4, 1

**— J —**

Jyh-Wei, Lee: TS1-2-MoA-9, 2

**— K —**

Kao Cheng, TAI: TS1-2-MoA-9, **2**  
Kazda, Tomas: TS1-2-MoA-10, 2  
Keckes, Jozef: TS1-2-MoA-4, 1  
Klemberg-Sapieha, Jolanta: TS1-2-MoA-3, 1

**— L —**

Lai, Yen-Chin: TS1-2-MoA-8, **1**  
Lee, Jyh-Wei: TS1-2-MoA-8, 1  
Lienard, Francois: TS1-2-MoA-4, 1  
Lou, Bih-Show: TS1-2-MoA-8, 1

**— M —**

Martinu, Ludvik: TS1-2-MoA-3, 1

**— P —**

Pitonakova, Tatiana: TS1-2-MoA-10, 2

**— R —**

Rada, Tomas: TS1-2-MoA-10, 2  
Rettenwander, Daniel: TS1-2-MoA-4, 1

**— S —**

Simek, Antonin: TS1-2-MoA-10, 2  
Soucek, Pavel: TS1-2-MoA-10, **2**

**— T —**

Tkadletz, Michael: TS1-2-MoA-4, 1  
Todt, Juraj: TS1-2-MoA-4, **1**

**— V —**

Vasina, Petr: TS1-2-MoA-10, 2

**— W —**

Wang, Chih-Liang: TS1-2-MoA-6, **1**